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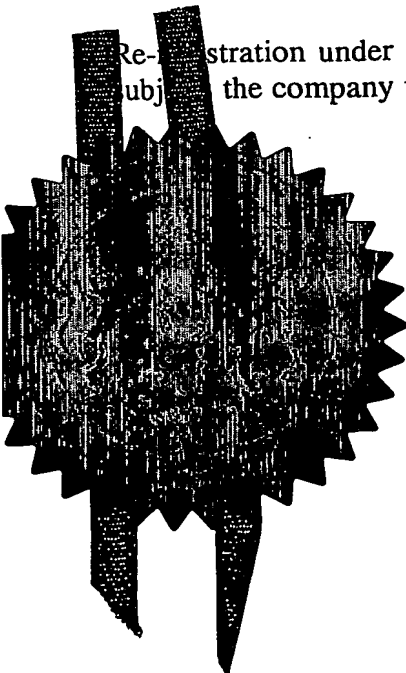
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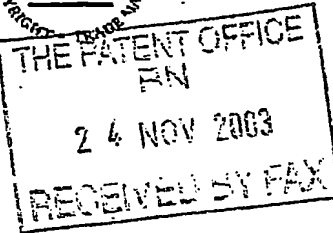
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3191

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0327267.1

## 3. Full name, address and postcode of the or of each applicant (underline all surnames)

University of Southampton

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79847.0001

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

## 4. Title of the invention

Fabrication of Zinc-diffused Waveguides in  
Periodically Poled Lithium Niobate

## 5. Name of your agent (if you have one)

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"Address for service" in the United Kingdom  
to which all correspondence should be sent  
(including the postcode)

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Signature(s)

*Alkene*

Date 24/11/03

12. Name, daytime telephone number and e-mail address, if any, of person to contact in the United Kingdom

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3191

## Fabrication of Zinc-diffused Waveguides in Periodically Poled Lithium Niobate

### 1 Review of Zinc-diffused Waveguides in Lithium Niobate

Quasi-phased-matched (QPM) wavelength conversion devices based on Lithium niobate waveguides have been widely studied for many years. The main applications of this technology include telecommunication systems, nonlinear optics, blue laser sources for next generation DVDs, etc. [1,2,3]. Several methods are now widely used for fabricating waveguides in this material, the most popular of which are annealed-proton exchange (APE) [4,5,6], Titanium diffusion, and ion implantation. However, each of these developed techniques has some limits of applicability [7].

Fabrication of waveguides on PPLN substrates generally involves two steps in a sequence that depends on the choice of waveguide formation technology. For example, proton exchange involves firstly poling the lithium niobate with a certain period of grating and then formation of channel a waveguide, whereas titanium diffusion reverses the order. Annealed-proton-exchanged waveguides are formed at relatively low temperatures (350°C-400°C) [5,6], so normally the waveguides are fabricated after the sample has been poled (poling after the waveguide formation results in a poor periodic grating structure). APE waveguides show increased resistance to photorefractive damage, however they support only extraordinary guided modes [5]. In addition, proton exchanged layers decrease the nonlinear coefficient in the initial proton exchange layer [6], requiring complex post annealing to recover the nonlinearity. An alternative low temperature technique, ion implantation, requires the use of ion accelerators and so is complex and expensive.

The normal temperature for Ti diffusion into  $\text{LiNbO}_3$  is around 1050°C to 1100°C [7], this is the process used to fabricate Ti-diffused  $\text{LiNbO}_3$  waveguides to be used in conventional optical components (eg: optical modulators, etc) which demonstrate good electro-optic properties, low propagation losses, and support both TE and TM modes [7]. However, the process is not compatible with periodically poled materials, because at such high temperatures the periodically switched domain structure is degraded. The alternative sequence process, poling after the formation of waveguides has been used with some success, but the formation of an unwanted thin domain inverted layer during the high temperature process for Ti diffusion may cause problems in the subsequent poling. An additional weakness of this technique is the worsened photorefractive damage in  $\text{LiNbO}_3$  induced by the incorporation of  $\text{Ti}^{4+}$  ions, which limits the operation of the  $\text{Ti}:\text{LiNbO}_3$  based devices to the infrared and visible range of the spectrum [7,8]. Several methods for suppressing out-diffusion have been proposed, for example surface polishing off the 50nm out-diffused layer after thermal processing, etc [8], but those steps add complexity to the technique. In addition, it is very difficult to provide a uniform periodically poled lithium niobate (PPLN) structure through a Ti in-diffused waveguide due to the electrical insulating properties of the lithium out-diffused layer in the waveguide area [7,8].

To overcome such difficulties, low temperature diffusion (below 1000°C) is desirable, requiring the use of elements with high diffusion coefficient and low activation energy. In this case, Zn appears to be a good choice [9]. In fact, low loss optical waveguides in  $\text{LiTaO}_3$  and  $\text{LiNbO}_3$  by Zn diffusion from the vapor phase have been demonstrated previously [9,11]. There is also a report on the fabrication of Zinc-diffused waveguides in y-cut  $\text{LiNbO}_3$  for 1.32 $\mu\text{m}$  wavelength operation by diffusing metallic Zn for applications to

electro-optic (EO) devices [10], but this has not been previously investigated for the z-cut crystal geometry necessary for electric-field-poled PPLN. The formation of Zinc waveguides in PPLN substrates grown by the Czochralski method (where the PPLN domains are formed into the crystal as it is grown) have also been made by Zn-diffusion from vapor phase in low temperatures [12]. However, fabrication techniques for the fabrication of Zn-diffused waveguides on PPLN have not been fully established yet, especially for z-cut electric field poled lithium niobate. In particular it should be noted that the closest reports of Zn diffusion into Czochralski-grown PPLN use a geometry in which the pyroelectric charges do not degrade the poling characteristics, and for which the material itself is limited in terms of quality and commercial viability. In our case, our ability to use z-cut substrates is surprising, as the pyroelectric effect is strongest for the z-cut geometry.

In this report, we present fabrication of Zn-diffused waveguides on z-cut electric field poled PPLN by thermal diffusion of a metallic Zn film. Various fabrication conditions were investigated and optimised to obtain good quality waveguides for different wavelengths through characterisation of mode profiles, numerical aperture (NA), spot sizes etc.

## 2 Fabrication of Zn-diffused PPLN Waveguide

In order to create a standardised route towards creating high-quality Zinc-diffused waveguides in z-cut electric-field-poled PPLN crystals, two distinct fabrication routes were investigated. The first approach was based on the assumption that it would be possible to periodically pole the lithium niobate crystals after the Zinc indiffusion process (an approach analogous to that used in Titanium diffusion), the results of which are described below. Investigation of a novel route towards incorporating Zinc-diffused waveguides into lithium niobate crystals after they have been poled forms the basis of the remainder of this patent application, allowing high-yield high-quality non-photorefractive devices to be fabricated using a completely new process.

During our first fabrication investigation, initial attempts to periodically-pole high-temperature Zn-diffused  $\text{LiNbO}_3$ , proved unsuccessful owing to the presence of a thin  $\text{Li}_2\text{O}$  out-diffusion induced domain-inverted layer on the +z face which is similar with the case for Ti-diffused  $\text{LiNbO}_3$ , this thin layer will block the electric field poling.

Figure 1 show the poling result of wet etching by diluted  $\text{HF:HNO}_3$  (1:2 by volume) solution at room temperature, from this figure we can see that the Zn-diffused area is under poled while the other area is completely over poled, the grating in the waveguide area is poor. From Figure 1, it is clear that poling after diffusion is not a good solution, so in this work, we adopted a novel method of fabrication Zn Waveguide on PPLN which involves poling the sample first, fabricating Zn-diffused waveguide afterward.

### 1.1 High electric Field Poling of Lithium Niobate

A z-cut 500- $\mu\text{m}$  thick, congruent  $\text{LiNbO}_3$  crystal is first cleaned in an ultrasonic bath at a temperature of 50  $^\circ\text{C}$ , then thin layer of photoresist (S1813) was spin-coated onto the -z face on which we defined the periodic pattern ( $\Lambda = 6.50\mu\text{m}$ , 10 $\mu\text{m}$ ) by using a Karl Suss MA4 mask aligner.

After developing the photoresist, gel electrodes were applied to both surfaces at room temperature. The samples were poled using a computer-controlled high electric field supply device that dynamically varied the applied field in order to maintain a designated current

curve, the poling process terminated when a predefined charge  $Q$  ( $=2AP_s$ ,  $A$ : opening Area;  $P_s$ : Spontaneous polarisation, for  $\text{LiNbO}_3$ ,  $P_s=0.72\text{uCmm}^{-2}$ ) had passed through the crystal. The applied electric field is around  $22.4\text{ kV/mm}$ , which is the appropriate value for domain reversal in  $\text{LiNbO}_3$ . The time duration of the poling is proportional to the calculated charge value which depends on the area to be poled. After poling, the sample was inspected under a microscope using crossed polarizers in order to check the quality of PPLN.

It should be noted that an additional consideration important to this procedure is that of producing high-quality periodically poled structures in lithium niobate crystals in the first place. During our recent experiments into the mechanics and optimisation of periodically poled materials, we have discovered that the highest quality and highest yield PPLN gratings are achieved when very thin periodic patterns are applied. For example, it is far simpler to fabricate a 'perfect'  $10\text{-}\mu\text{m}$ -wide PPLN grating over a distance of centimetres than it is to create a  $1\text{-mm}$ -wide grating (an effect likely due to avoiding as many defect sites in the crystal as possible). This effect also applies to the achievable periods in the sample, as the increased quality of thinner gratings allows finer periods of less than  $10\mu\text{m}$  to be readily achieved, which is also more difficult in wider gratings. In the case of fabricating channel waveguides in PPLN for high-efficiency second-harmonic-generation, it is often the case that such short period gratings are required.

One of the primary benefits of our investigation into creating a process that allows us to introduce waveguides into already poled PPLN samples is that the poling yield can be optimised before the waveguide fabrication takes place. This is important as any changes to the surface of the material, such as the incorporation of Zinc or out-diffusion of Lithium, can drastically change the poling characteristics later on. As typical waveguide dimensions are  $10\text{-}\mu\text{m}$ -wide or less, a further benefit of this process is that the metallic strips necessary for Zinc diffusion into the material can be precisely defined on top of existing PPLN gratings to ensure perfect alignment and therefore maximum device efficiency. When the two techniques of thin grating poling and Zinc-diffusion are combined, this provides an excellent route towards high-yield device fabrication.

### *1.2 Zn Diffused Waveguide on PPLN by Thermal Diffusion Technology.*

**After Poling.** The lithium niobate samples were thoroughly cleaned to remove any surface particulates that could cause contamination and influence the thermal diffusion process. Zn-diffused channel waveguides strips spaced  $100\mu\text{m}$  apart and oriented parallel to x-direction, with widths varying from  $1.0\mu\text{m}$  to  $7\mu\text{m}$  were patterned by photolithography. Because Zn atoms adhere poorly to the surface of  $\text{LiNbO}_3$ , [10], a  $5\text{-}10\text{nm}$  thick Ni film was deposited onto the  $-z$  face of the PPLN substrate prior to the Zinc in order to increase the adhesion. Zinc films with thickness varying from  $80\text{nm}$  to  $150\text{nm}$  were then evaporated onto different samples using a standard lift-off technique. After patterning the metal layer, the samples were placed into a covered platinum crucible in a high-temperature oven.

Here, the thermal in-diffusion cycle involves heating the crystal at a rate of  $6\text{ }^\circ\text{C/min}$  to diffusion temperature, maintaining that temperature for certain time, then cooling back down to the room temperature at rate of  $6\text{ }^\circ\text{C/min}$ . In our experiments all the in-diffusion processing was carried out in dry air. The different samples and diffusion conditions are listed on Table 1.

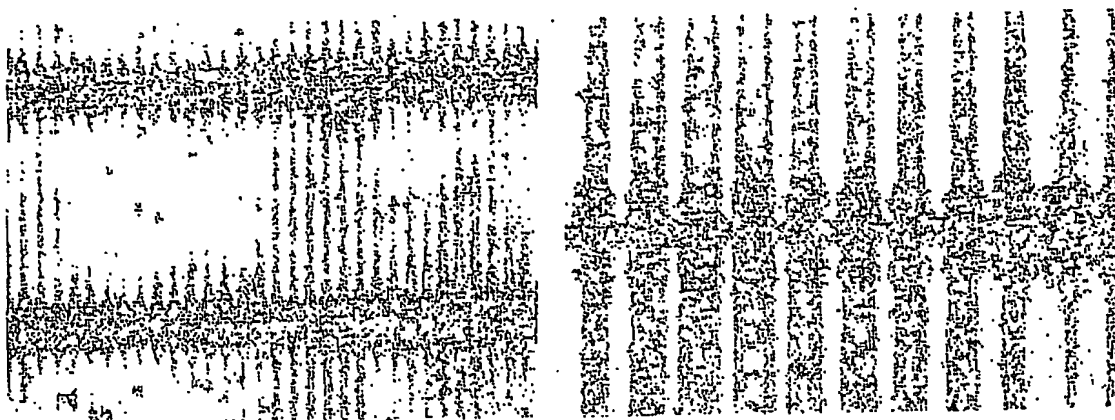


Figure 1. Poled after thermal diffusion. Periodical poling grating on Zn-diffused waveguide under the microscope after wet etching, the period of the PPLN is 10 $\mu$ m, the width of waveguide is 8 $\mu$ m.



Figure 2. Poled before thermal diffusion. Zn-diffused waveguide on PPLN under the microscope, The period of PPLN is 6.5 $\mu$ m and the waveguide is around 3.6 $\mu$ m.

Figure 3. Poled before thermal diffusion. Zn-diffused waveguide on PPLN under the microscope after wet etching. The period of PPLN is 10 $\mu$ m and the waveguide is around 8 $\mu$ m.

In order to make sure that the PPLN is still present after these high temperature treatments, one sample (No.2 on Table 1) was etched in a solution of HF:HNO<sub>3</sub> (1:2) for 10 minutes. Figure 2 shows the poling before the thermal Zn diffusion of the waveguide on lithium niobate under the microscope and Figure 3 shows the wet etching result of a 6 $\mu$ m wide Zinc-diffused region on PPLN with a 10  $\mu$ m period grating. Comparison of the figures demonstrates the uniformity of the preserved grating over the entire length of the poled sample, and thus the problem of the random fluctuations in domain wall position is overcome. This result confirms that the periodically inverted domain structure of the PPLN substrate maintains its original form during the diffusion processes.

After waveguide fabrication, the end faces of the sample were cut and polished to allow optical characterisation. The final device length was approximately 12mm.

### 3. Characterisation of Zn-diffused Waveguide on PPLN

Characterisation of the Zinc-diffused channel waveguides was carried out using a Ho-Ne laser ( $\lambda = 632.8\text{nm}$ ) fibre-butt coupled into the channel waveguides. A 10x lens was used to focus the output beam onto a CCD camera mounted in a translation stages which can be moved along the beam propagation direction so that the near and far field modes can be recorded, and the CCD camera was linked to a PC which allows the measurements of spot size of the waveguide, mode profile and numerical aperture (NA) etc.

Table 1. Result of characterisation for waveguide under different diffusion condition

No	Time (min)	Thickness (nm)	Diffusion Temp (°C)	NA-Y	NA-Z	Spot size-y (µm)	Spot size-z (µm)
1	80	80	930	0.079	0.105	4.09	3.26
2	120	80	930	0.074	0.094	4.74	3.51
3	150	80	930	0.064	0.082	6.34	3.71
4	180	80	930	0.050	0.080	6.87	3.96
5	80	80	900	0.093	0.104	4.34	3.11
6	120	100	930	0.080	0.109	4.43	3.23
7	120	120	930	0.089	0.115	3.81	3.14
8	120	150	930	0.090	0.120	3.49	3.06

Figure 4 shows the TM and TE mode profiles for channel waveguides in sample No 2. Here, the width of the deposited metal was 2.0µm, the modes are single mode (for wavelength of 633nm) with full width at half maximum (FWHM) mode size of 4µm in y direction and 2.5µm in Z direction for TM mode and 4.3µm in y direction and 3.5µm in Z direction for TE mode. Figure 4 indicates that Zn-diffused waveguide supports both TM and TE mode.

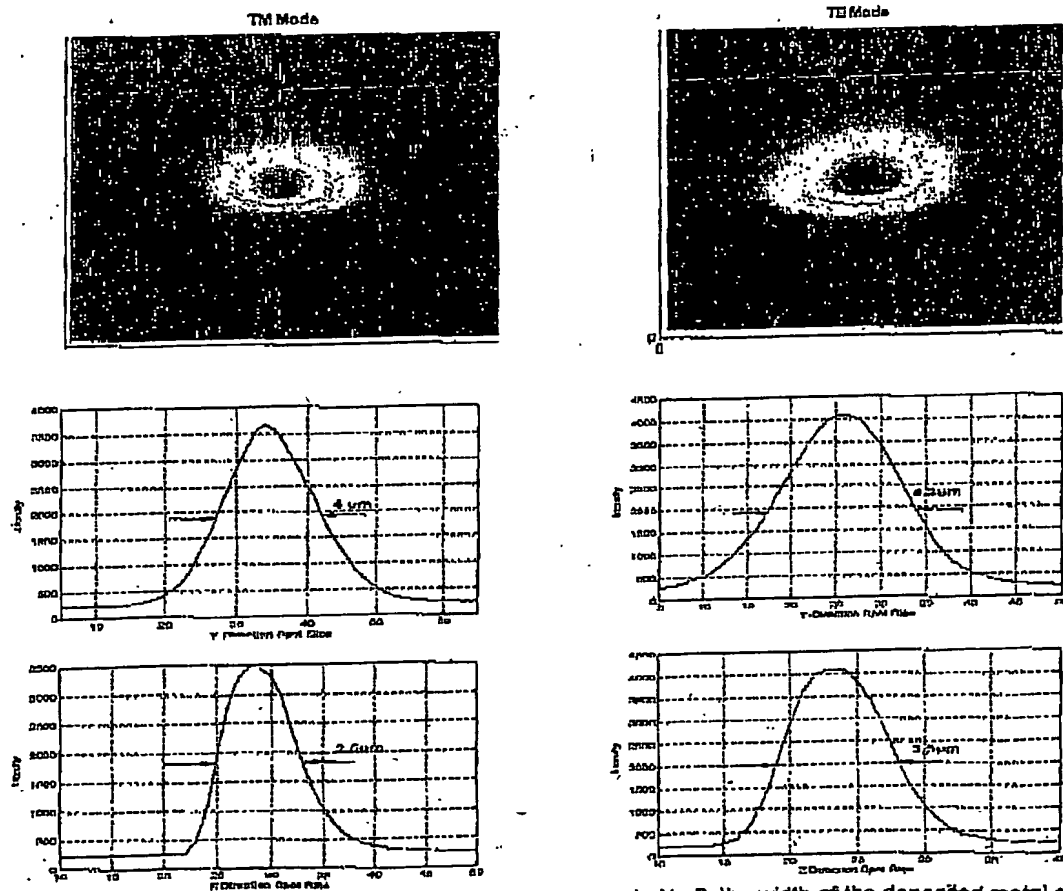


Figure 4. TM and TE mode profiles of waveguide on the sample No 2, the width of the deposited metal strip was 2.0µm, the mode is single mode with spot size of 4µm in y direction and 2.5µm in Z direction for TM mode and 4.3µm in y direction and 3.5µm in Z direction for TE mode.

The far-field technique has been adopted to measure the NA of waveguide, figure 5 shows the dependence of the spot size of sample No.2 on the distance of the CCD camera to the focusing point, obviously larger distances lead to larger spots and in turn give a better estimate of the NA. When the distance  $D \gg$  core size of waveguide, the NA tends to be a constant figure which is 0.08 in y direction and 0.11 in z direction.

Based on the NA obtained, the refractive index change ( $\Delta n$ ) of sample No.2 has been calculated, the  $\Delta n$  is around 0.0015 in the y-direction and 0.0025 in the z-direction under the diffusion conditions listed in Table 1.

Figure 6 shows the dependence of the FWHM spot size and the NA as a function of diffusion times that is varied from 80min to 180min. In all cases, the thickness of Zn films was 80nm and the diffusion temperature was 930 °C. The longer the diffusion time, the bigger spot size and the smaller the NA obtained. It is observed that the spot size change in the z direction is smaller than it is in the y direction, the reason for the dependence is being studied. The dependence of the FWHM spot size and the NA on thickness of the Zn film was also measured and is shown in Figure 7, the samples with Zn film varied from 80nm to 150nm were diffused at a temperature of 930 °C for 120 min. Figure 7 indicates that a thicker Zn film results in a smaller spot size and a larger NA. Figure 8 shows the dependence of the width of single mode channel waveguides for different wavelengths as a function of the diffusion time at a diffusion temperature of 930 °C, where the Zn film was 120nm thick.

From Fig.7, it is clear that the thicker the Zn film is, the smaller mode spot size and better confinement of the waveguide, hence higher the power intensity within the waveguide. But thicker Zn film result in a rougher surface due to the Zn remnant (which was probably caused by Zn-LiNbO<sub>3</sub> reaction in the air) after the thermal indiffusion processing, hence higher propagation loss due to the scattering of light in the waveguide surface, it was also found that when Zn film is 150nm, the remnant is far more rough than that of 120nm.

From figure 5 to figure 8, It is clearly shown that fabrication channel waveguides for different wavelengths requires different diffusion conditions, samples with thicker Zn film diffused at a shorter time is favourable for the fabrication of single mode channel waveguides with strong confinement and small spot size, thinner Zn film and longer duration time is suitable for single-mode channel waveguides with smaller NA and bigger spot size. The trade-off of diffusion conditions of fabrication single mode channel waveguides for wavelength of 980nm, 1064nm and 1550nm are listed in table 2.

Table 2. The trade off diffusion conditions of Waveguide fabrication for different wavelengths.

Wavelength (nm)	Thickness of Zinc layer (nm)	Diffusion Time (min)	Diffusion Temperature (°C)	Deposited Zinc strip width (μm)
980	80	80	930	3.2
1064	80	120	930	3.6
1550	120	120	930	6.4

A Nd:YAG laser at 1064nm was then used to study how effective the "poling before Zn diffusion" approach would be in achieving uniformity of the periodically switched domain structure inside the Zn waveguide after the thermal diffusion. High quality domain gratings are required for quasi-phase matched second harmonic generation (SHG) in waveguides. The incident beam was TM polarized in order to take advantage of the optimal non-linear susceptibility tensor coefficient  $d_{33}$ . An end polished waveguide sample with PPLN of 6.5μm period and a Zn waveguide of width 3.6μm (the diffusion conditions as listed in Table

2) was put on a temperature tuneable oven, the output beam was focused into an optical power meter, the power of SHG green light (532nm) from Zn-diffused waveguide on PPLN was measured based on different temperature. The quasi-phase matching curve dependent on tuning temperature shown in figure 9 indicates the QPM temperature is around 145.1 C°, the maximum output external SHG power of 150uW was recorded for a internal pump power of 30mW of fundamental beam, which corresponds to a conversion efficiency of 16.7%/Wcm<sup>2</sup> with the waveguide length of 10mm.

The measurement was carried out for 3 hours and no photorefractive damage or SHG output power fluctuations were found. This indicated that the influence of photorefractive effect induced by generation of the second harmonic beam was depressed due to the Zn diffusion waveguide.

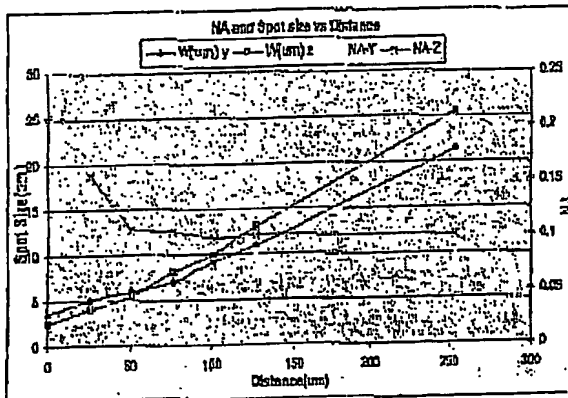


Figure 6. Dependence of spot size on distance of CCD camera to focusing point

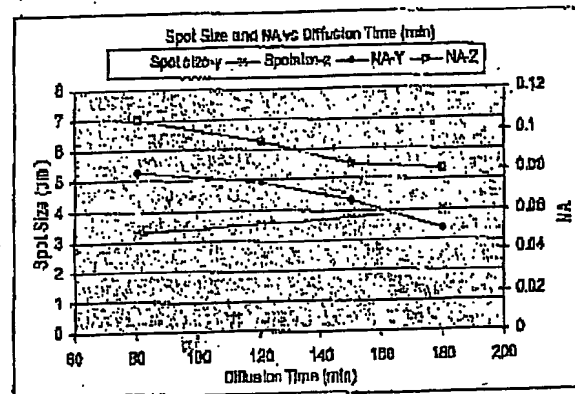


Figure 6. Dependence of FWHM spot size and NA on diffusion times.

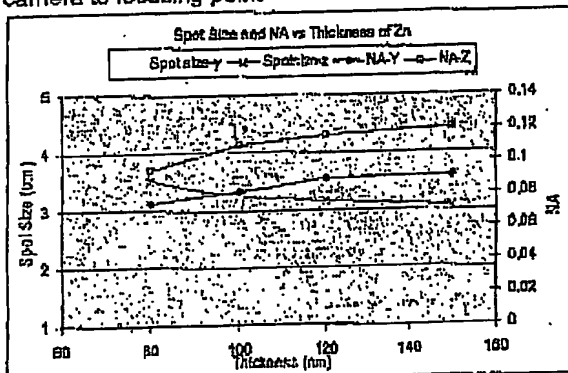


Figure 7. Dependence of FWHM spot size and NA on thickness of the Zn film

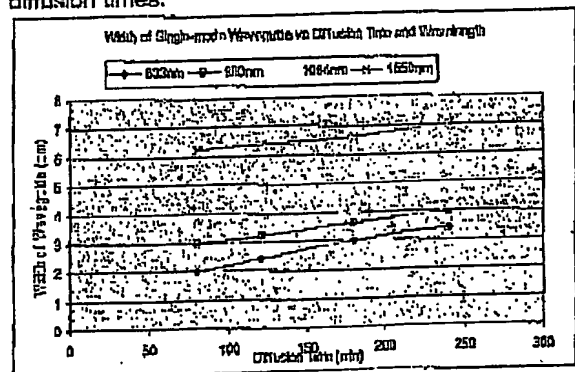


Figure 8. Dependence of width of single mode waveguide for different wavelengths on the diffusion times.

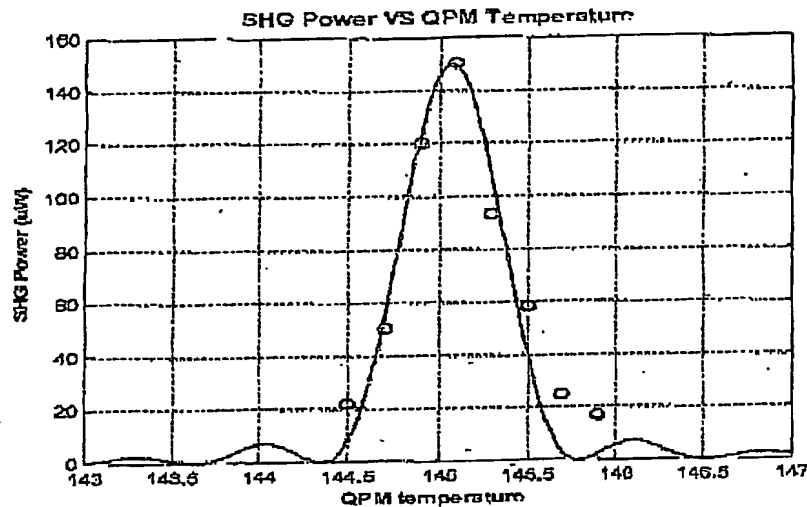


Figure 9. Dependence of SHG output power on the QPM temperature. The continuous line is calculated, the spots indicate experiment data.

#### 4. Discussion of Advantages of Zn-diffused Waveguides in PPLN

In comparison to Ti-indiffused and annealed proton exchanged waveguides in Lithium Niobate, Zn-diffused waveguides have following advantages:

- **High resistance to photorefractive damage:** Zinc-based waveguides are expected to allow efficient operation at room temperature and blue/visible operation, both of which are unattainable via Ti-diffusion or Proton Exchange.
- **High-yield periodic poling before diffusion:** Zn-diffusion is carried out at a lower temperature than Ti-diffusion (930°C rather than 1050°C) allowing the periodically-poled structures and nonlinear coefficient  $d_{33}$  in the sample to be completely preserved. This is a huge advantage as poling can be performed in virgin wafers using pre-optimised parameters for a high yield, and with no diffusion-related issues of material modification.
- **Support both TE and TM modes:** The Zn diffused waveguides support both TE and TM modes, a result unattainable via Proton Exchange.
- **High production efficiency:** As the diffusion time for Zinc diffusion into lithium niobate is far shorter than that for Ti diffusion or annealed proton exchange [6,7], high volume production efficiency will also increase accordingly.
- **Novelty:** Zinc-diffusion into z-cut PPLN is a completely new technique that has yet to be exploited. Ti-diffusion and Proton Exchange (despite their various drawbacks) are well established at various facilities worldwide, particularly Paderborn and Stanford Universities.

#### 5. What we claim in this invention.

1. Fabrication of PPLN by high electric field poling by Gel electrode.
2. Zn diffused waveguide in Z cut LN by thermal metallic diffusion.
3. Zn diffused waveguide by thermal metallic diffusion in Z-cut PPLN made by high electric field poling.



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